INFRARED SPECTRUM ANALYSIS OF THE HYDRO-CHLORIC ACID-WATER-CARBON TETRACHLORIDE SYSTEM

by

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THESIS

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ABSTRACT

The infrared vibrational-rotational spectra of hydrochloric acid and water dissolved together in carbon tetrachloride were obtained and compared with the spectra of hydrochloric acid in carbon tetrachloride and of water in carbon tetrachloride separately. Spectra of extremely dilute solutions of the two solutes in the non-polar solvent were analyzed for nature and extent of solvent-solute interaction. Dilution behavior was investigated by the use of the Beer-Lambert Law. Possible resulting species were also investigated.

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I. INTRODUCTION

A. GENERAL

The infrared spectrum, which can arbitrarily be designated the region between two to fifty microns of wavelength, yields information concerning vibrational and rotational energy levels and the transitions in vibrational-rotational energy. If a molecule is to absorb infrared radiation, the radiation must be of the correct frequency to cause a quantum jump in its vibrational-rotational energy. A vibrating molecule will interact with electromagnetic radiation if the vibration is accompanied by an oscillating dipole moment which occurs whenever a change occurs in the position of the centers of positive and negative charge resulting from atomic motion.

Infrared spectra of simple molecules such as hydrochloric acid and water in non-polar media can be studied to yield information regarding the interaction of a chemical species with its environment. Extremely dilute solutions of water in carbon tetrachloride and in carbon disulfide and dilute solutions of hydrochloric acid in carbon tetrachloride yielded spectra with marked differences from spectra of the pure components [1-5]. These differences yielded information on the extent and nature of the interaction between solute and solvent. This investigation was to study the molecular structure and various parameters such as force constants and vibrational frequencies of hydrochloric acid in carbon tetrachloride by means of infrared spectroscopy. Hygroscopicity of the solvent and of the $HCl-CCl_4$ solutions led this investigation to an analysis of the interactions of a three-component system, hydrochloric acid-water-carbon tetrachloride.

B. INFRARED SPECTRA OF HYDROCHLORIC ACID

The infrared spectra of hydrochloric acid (HCl) has been intensively studied in the vapor phase, liquid phase, and in dilute solutions of non-polar solvents. The vapor phase and liquid phase spectra at various pressures and temperatures were studied in considerable detail in 1939 by West [6].

The HCl molecule is a linear heteronuclear diatomic molecule which is an example of $C_{\infty V}$ symmetry and therefore possesses rotational- vibrational fundamentals which are active in both infrared and Roman spectra [7]. The internuclear distance for the molecule at equilibrium at 25°C has been reported to be 1.2747×10^{-8} cm [7]. Hettner has presented the vibration frequencies (fundamentals) of HCl in the gaseous and liquid states as 2885.9 cm^{-1} and 2785 cm^{-1} respectively [8]. The fundamental absorption band of HCl is measured at the band center since Q-branch transitions (for which $\Delta J = 0$) do not occur for HCl in its pure state.

C. INFRARED SPECTRA OF HCl IN NON-POLAR SOLVENT

When HCl, either in gaseous form or liquid form, was diluted with non-polar solvent such as carbon tetrachloride, its fundamental vibration frequency was shifted as reported by Lascombe, Van Huong, and Josien [9] and by Jones and Sheppard [20]. A new absorption band was reported and this new absorption was investigated by Iohansen and Broun [10] who called it the Q-branch of the hydrogen halide spectrum. Iohansen concluded that the central band in the HCl spectrum was the band of the fundamental vibration in a complex Cl-H···Cl with a hydrogen bond, and the secondary bands belong to free HCl molecules. Barrow and Datta [11] have reported far-infrared spectra of HCl and DCl in carbon tetrachloride and they have observed that the separation of the rotational and the

vibrational motions can be assumed for condensed systems. Bulanin, Orlova and Shchepkin [12] have shown that the IR band shapes are influenced greatly by the rotational motions of molecules in liquid solutions, not necessarily non-polar.

D. INFRARED SPECTRA OF HOH

The infrared spectrum of water has been intensively studied in vapor phase, liquid phase and in dilute solutions of non-polar solvents. The vapor phase spectrum was first analyzed in considerable detail by Darling and Dennison [13]. The water molecule is known to have a C_{2v} symmetry and therefore possesses three normal modes of vibration which are all active in both the infrared and Raman spectra. Benedict, et al. give the values of the three fundamental vibrational frequencies of water vapor as: $\nu_1 = 3656.65\ cm^{-1}$, $\nu_2 = 1594.56\ cm^{-1}$, $\nu_3 = 3755.79\ cm^{-1}$ [14].

Liquid water shows the characteristic very broad, intense infrared absorption band around 3400 cm^{-1} found for OH containing compounds and attributed to associated or hydrogen bonded species [15].

E. INFRARED SPECTRA OF HOH IN NON-POLAR SOLVENTS

When HOH was dissolved in non-polar solvent such as carbon tetrachloride, a shift in its fundamental frequencies was observed and these new absorption peaks were assigned to the stretching frequencies of the unassociated HOH molecule [1-3]. Fox and Martin assigned the observed absorption at 3705 cm^{-1} as ν_3 and at 3614 cm^{-1} as ν_1 [1] and this was confirmed by Riesinger in his investigation [4]. ν_2 was not observed for investigations in carbon tetrachloride since the solvent absorbs intensely in the region expected for that fundamental frequency. Morris further

investigated the additional bands at 3804 cm^{-1} and at 3540 cm^{-1} and concluded that they were envelopes of rotation lines which indicated that the water molecules were relatively free to rotate [5].

II. EXPERIMENTAL PROCEDURE

The Perkin-Elmer Grating Infrared Spectrophotometer Model 621 was used in the double beam mode of operation. A general calibration was accomplished using the 0.05 mm polyethylene standard window and the critical frequencies checked out as published. Initial samples were placed in 1-mm path sodium chloride cells. Insufficient absorption data was obtained due to the low concentration of solute in the carbon tetrachloride solvent.

Connecticut Instrument Corporation 50-mm path length glass cell with barium fluoride windows was next used. This cell has a capacity of approximately 31.5 milliliters. The absorption spectra obtained with this cell proved to be too intense even at extremely dilute solutions.

Barnes Engineering Company 10-mm path length glass cell was used with barium fluoride windows. This cell has a capacity of approximately 7.0 milliliters. Only glass, Teflon washers and the barium fluoride windows were in contact with the sample in the cell. The absorption spectra obtained with this cell provided the necessary data for the investigations.

The carbon tetrachloride used as solvent was Fisher Scientific Company Spectranalyzed grade reagent. No additional purification other than drying the solvent was necessary. Drying techniques used in this laboratory followed procedures suggested by Morris in his investigation [5]. Union Carbide (Linde Company) Type 4A molecular sieve in bead form was placed in a separatory funnel with the solvent. A 50-ml burette was also filled with molecular sieve as a second drying column before using the solvent. The disappearance of the ν_3 and ν_1 fundamental water

frequencies was used as the criterion for dryness of the solvent. Contact with atmospheric moisture was kept to a minimum by rapid handling and by filling containers directly from the drying column.

Samples of the hydrochloric acid used were in gaseous form which was available in compressed tanks from Air Products and Chemicals, Inc. and in liquid concentrated form which was C. P. Reagent grade (37% - 38% HCl) from Allied Chemical Company.

Use of concentrated HCl solutions presented disadvantages due to inherent presence of water in high concentration and its relatively less solubility of the HCl in the carbon tetrachloride solvent. The gaseous HCl was dried with calcium chloride and alumina prior to use.

Use of the gaseous HCl involved bubbling the gas through the solvent at room temperature. A 2-minute bubbling time through 100 ml of carbon tetrachloride generally yielded approximately 0.1 M of HCl in the solvent. This concentration proved to be too high for good absorption spectrum analysis and was therefore diluted to approximately 0.03 - 0.05 M solutions.

Determination of the HCl concentration in solution was accomplished by extraction with water, taking advantage of the relative solubilities of HCl in water and in carbon tetrachloride. After extraction, the HCl concentration was determined by standard titration techniques and also by means of a Beckmann pH meter. Both methods gave satisfactory results.

Determination of water concentration in the carbon tetrachloride was predicated upon published data by Clifford [16] and later used by Riesinger and Morris in their investigations. Riesinger determined that at least four hours waiting period was required for water to completely dissolve in carbon tetrachloride. A saturated solution was obtained by adding 30 lambdas of water to 100 milliliters of carbon tetrachloride and letting the solution stand overnight.

The DOD used during this investigation was obtained from BIO-RAD Laboratories and stated to be 99.83 percent DOD. The DCl solution contained 38 percent of DOD and was used for spectra comparisons with HCl solutions.

III. RESULTS AND DISCUSSION

A. SPECTRA OF WATER IN CARBON TETRACHLORIDE

Analysis of infrared spectra of dilute solutions of HOH and DOD in carbon tetrachloride confirmed results obtained by previous investigations [17,18]. The most recent work with which these results were compared was that of Morris [5]. The observed fundamental frequencies for HOH were ν_1 at 3614 cm^{-1} and ν_3 at 3705 cm^{-1}; both agreed with the reported values. Also observed were the envelopes of rotation lines of the HOH molecule at 3804 cm^{-1} reported by Fox and Martin [18] and at 3540 cm^{-1} reported by Riesinger [4]. The fundamental frequencies for DOD were ν_1 at 2642 cm^{-1} and ν_3 at 2751 cm^{-1}; both also confirmed values reported by Morris [5]. The ν_3 band (OH stretching) for HOD at 3660 cm^{-1} was observed when DOD in carbon tetrachloride solution was exposed to atmospheric moisture since the following reaction is known to be rapid [19]:

$$HOH + DOD \rightleftarrows 2\ HOD \qquad K_{eq} = 3.96 \text{ at } 25^{\circ}C$$

The OD stretching band for HOD was not observed since its absorption band had considerable overlap with the DOD spectrum at 2642 cm^{-1} and 2751 cm^{-1}.

B. SPECTRA OF HCl IN CARBON TETRACHLORIDE

Analysis of infrared spectra of dilute solutions of HCl and DCl in carbon tetrachloride confirmed results obtained by Bulanin, et al. [12]. The observed absorption frequency for the Q-branch at 2830 cm^{-1} for HCl and 2050 cm^{-1} for DCl were shifted from the gaseous absorption frequencies of 2886 cm^{-1} and 2090 cm^{-1} respectively. (Fig. 1) These

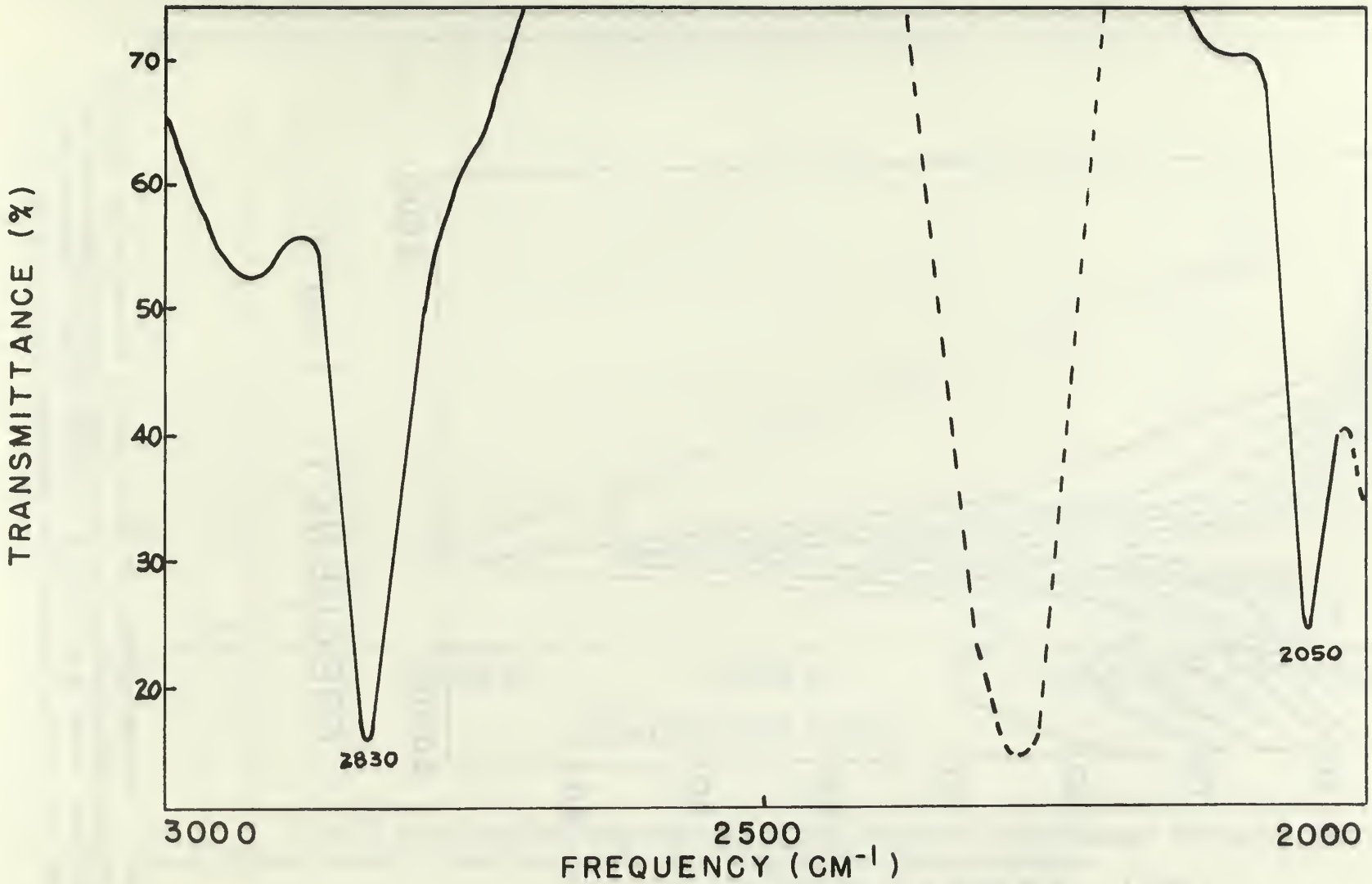

Figure 1. Infrared spectrum of HCl-DCl (~ 0.025M) in carbon tetrachloride solution. Absorption peak at 2830 cm^{-1} is due to HCl absorption and the peak at 2050 cm^{-1} is DCl absorption. Dotted lines are absorptions due to the solvent.

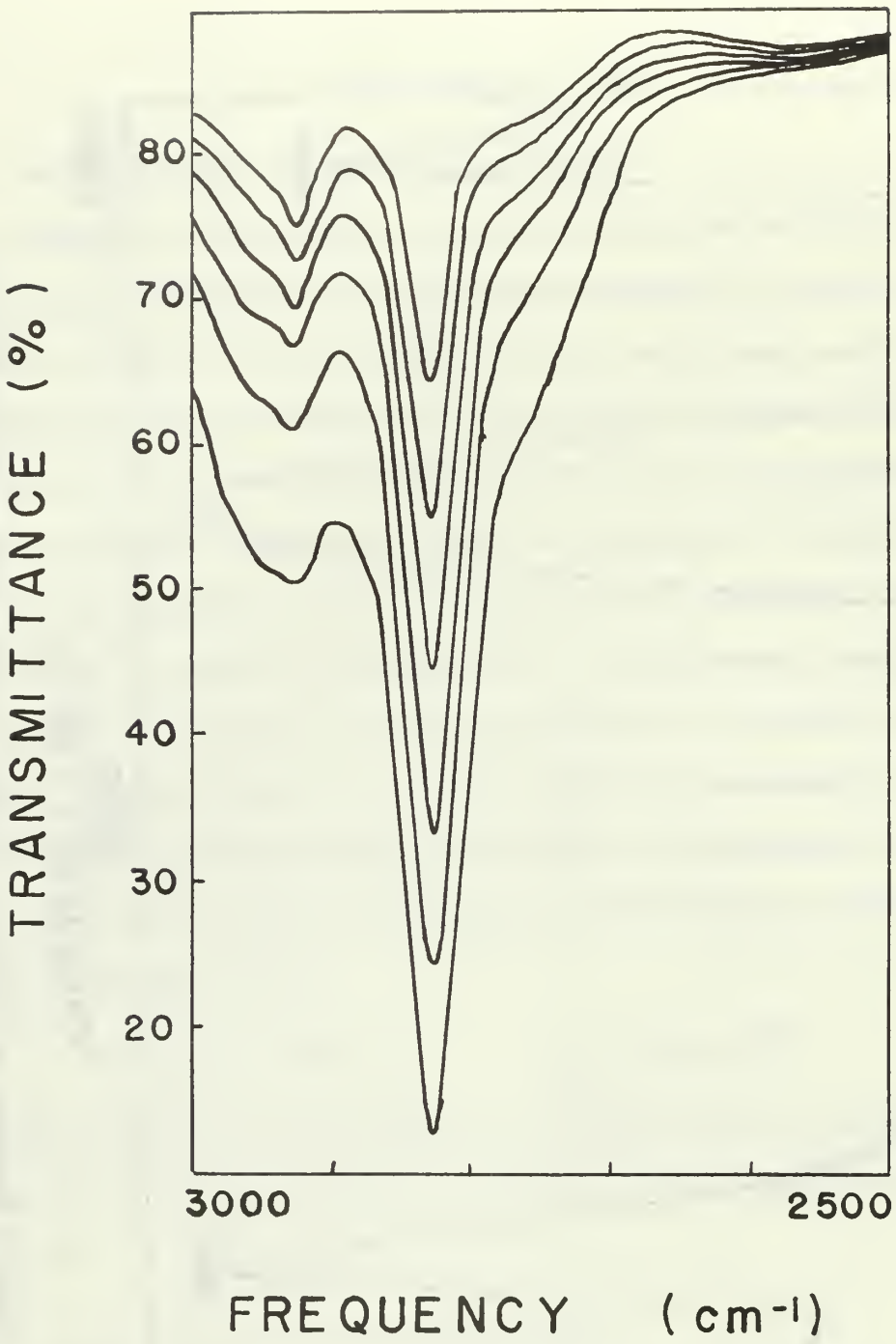

Figure 2. Infrared spectra of HCl in carbon tetrachloride solution, with varying concentrations of the solute. The HCl concentration ranged from ~ 0.001 M to 0.020 M, with the higher concentrations showing less transmittance at the absorbance peak (2830 cm^{-1}).

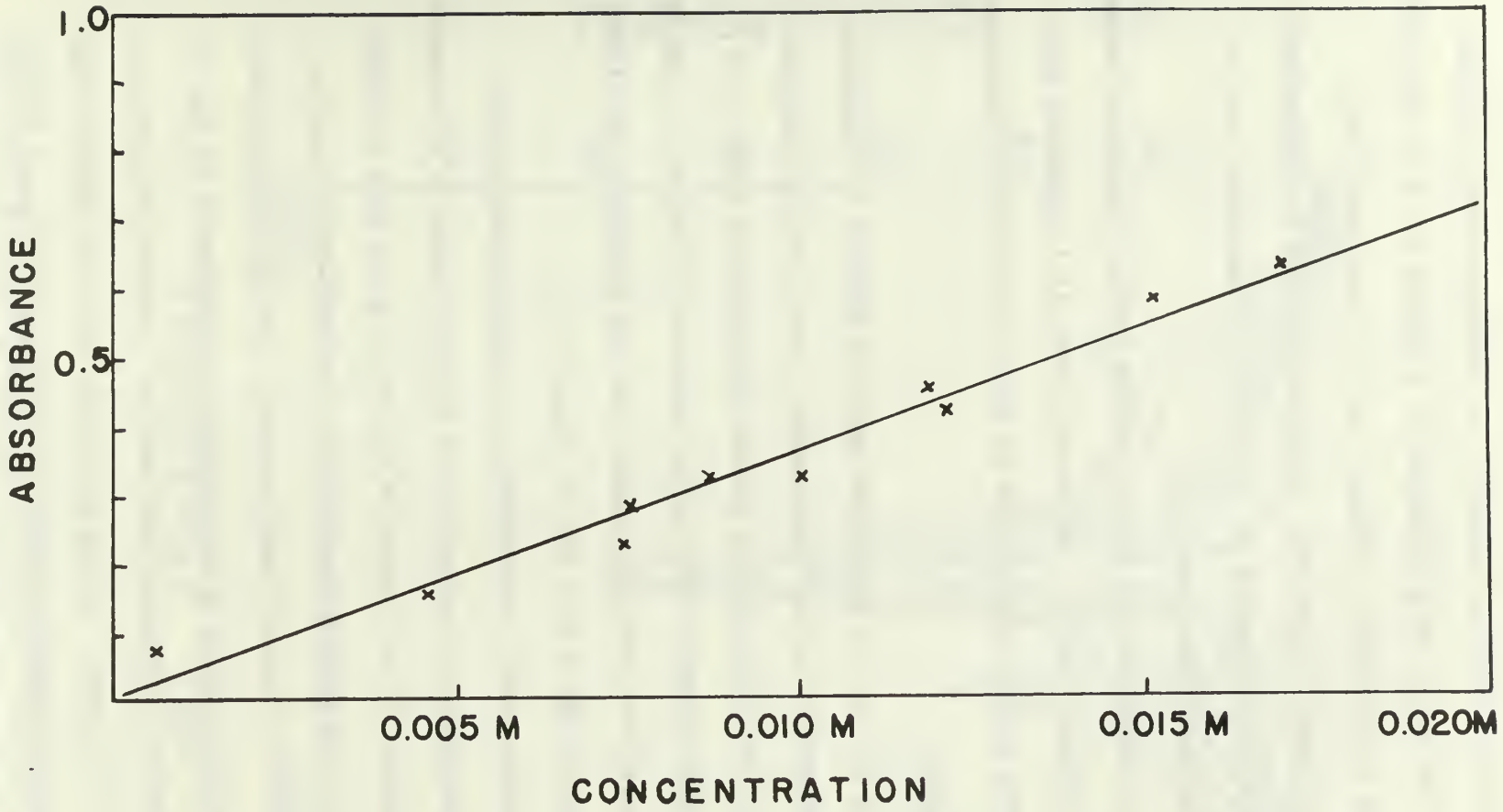

Figure 3. Plot of absorbance vs concentration of HCl in carbon tetrachloride solution. The values were obtained using a 1-cm path length cell with barium fluoride windows.

shifts of -56 cm^{-1} and -40 cm^{-1} strongly suggest the interaction of the solute with solvent molecules, primarily by hydrogen bonding between HCl and CCl_4. The shifts were different by a factor of $\sqrt{2}$ in agreement with theoretical calculations. Bulanin, et al., attributed the Q-component to the purely vibrational transitions of the solute molecule and the shoulders of the spectra as the P- and R- components due to rotational-vibrational transitions [12].

Far infrared studies of HCl in CCl_4 showed similar absorption bands as in the near-infrared region. Barrow and Datta [11] suggested that these far-infrared absorptions are due to purely rotational transitions.

C. DILUTION EFFECTS

The intensity of the HCl absorption peak was studied to determine whether the Beer-Lambert Law was applicable to the HCl-CCl_4 solution. The resulting plot of absorbance vs solute concentration (Figs. 2-3) shows a fairly good agreement with dilute solutions (0.001 M to 0.020 M). It was also observed that the Beer-Lambert Law was applicable to dilute H_2O-CCl_4 solutions (0.001 M to 0.022 M) (Figs. 4-5).

The determination of HCl concentration in carbon tetrachloride solution was accomplished by extraction of HCl with water in a stoppered flask. An approximate 50 to 1 ratio of H_2O to CCl_4 was used to obtain maximum extraction and minimum loss of HCl. The acid-water solution was then titrated with standard NaOH solution. The pH meter was also used to determine the acidity of the solution. Calibration of the pH meter with standard HCl solutions gave results to within 15% accuracy in the 10^{-1} to 10^{-4} M range. It was also found that the concentration of HCl in CCl_4 did not remain constant for more than 3-4 hours at 25°C. This made it absolutely necessary to determine HCl concentration for every spectra taken.

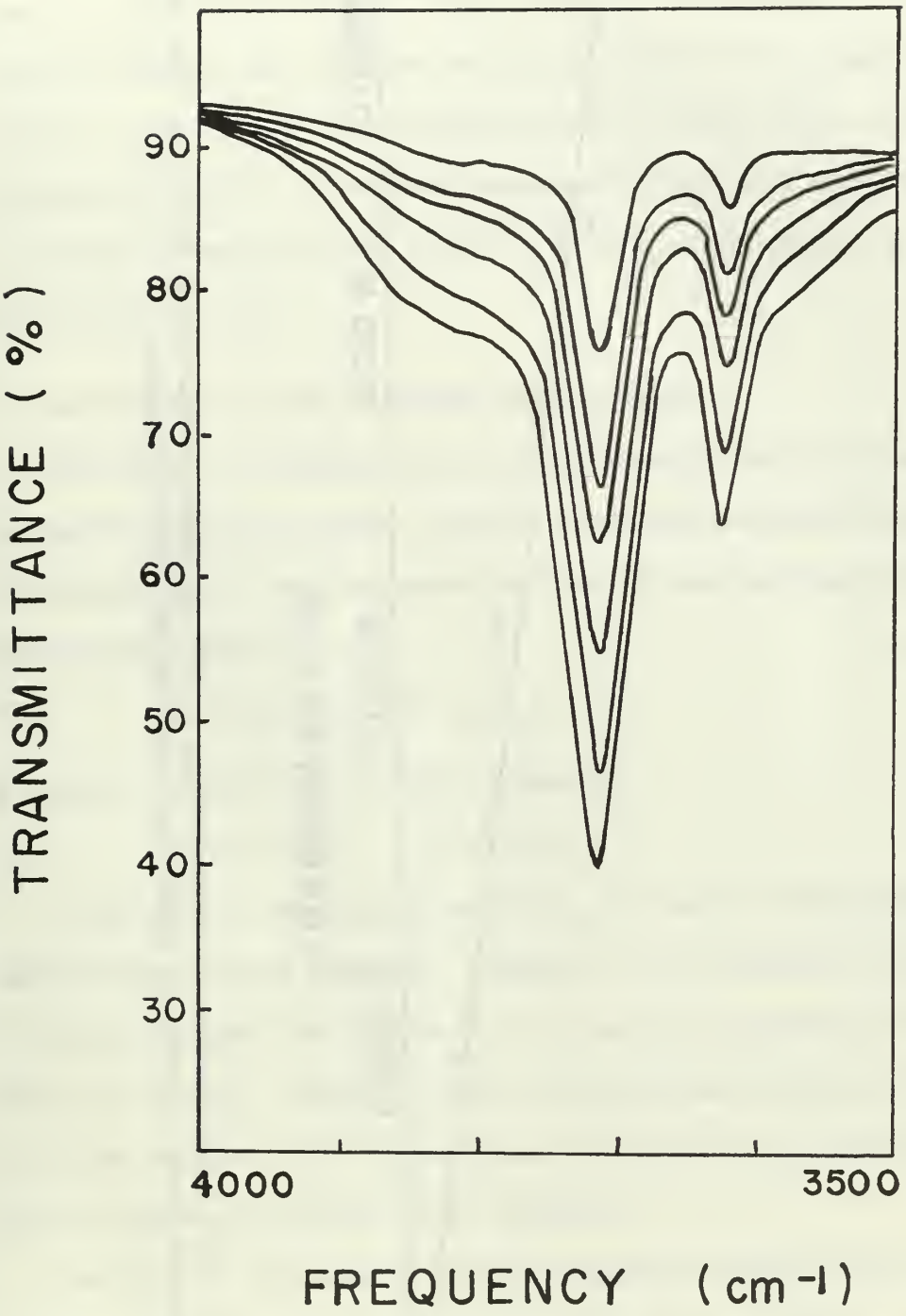

Figure 4. Infrared spectra of HOH in carbon tetrachloride solution, with varying concentrations of the solute. The HOH concentration ranged from 0.001 M to 0.022 M. The absorbance peak at 3705 cm^{-1} (assigned to the anti-symmetric OH stretching mode) shows the Beer's Law behavior of the solution.

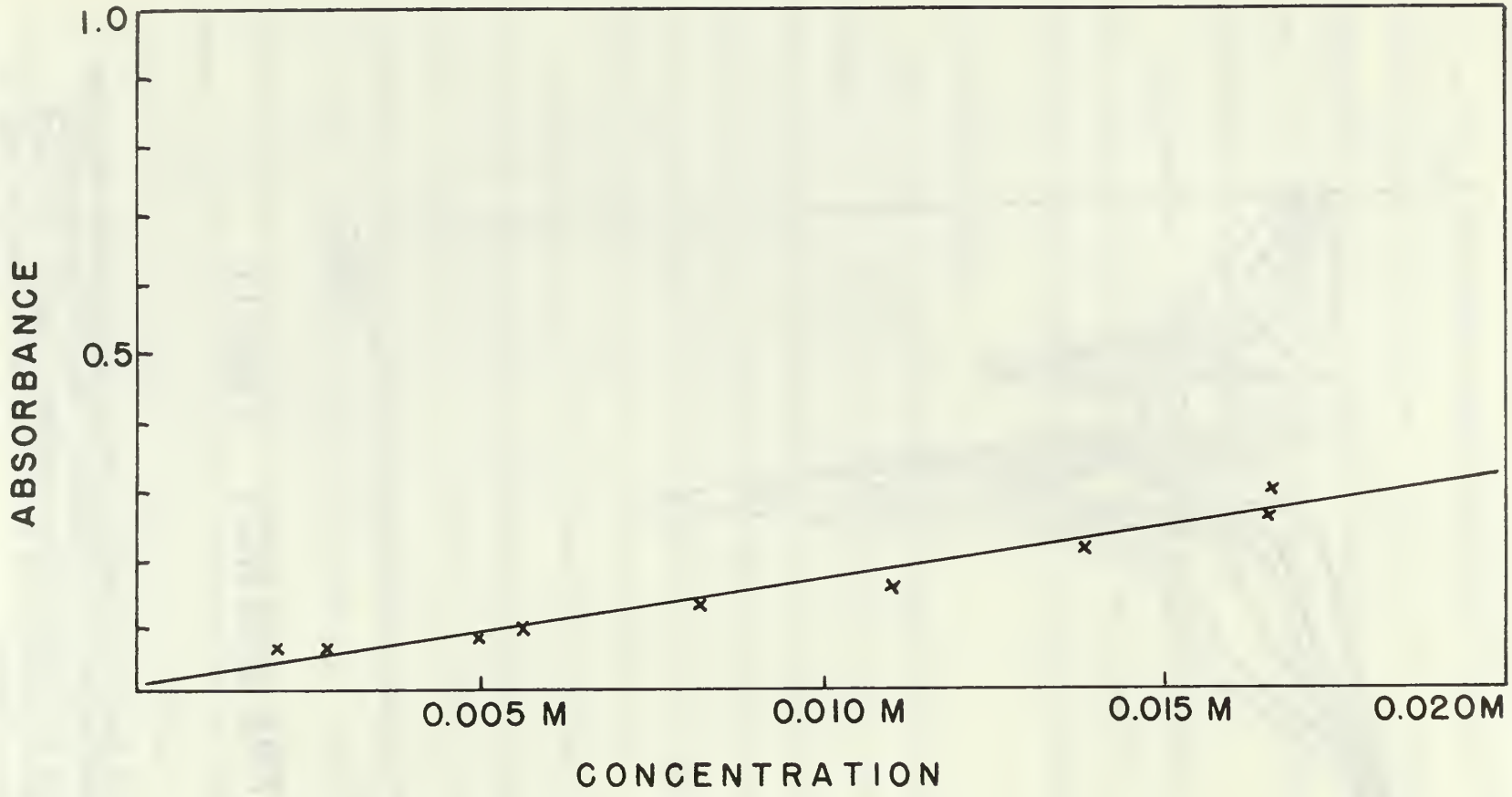

Figure 5. Plot of absorbance vs concentration of HOH in carbon tetrachloride solution. The values were obtained using a 1-cm path length cell with barium fluoride windows.

Calculation of the maximum HOH concentration in Carbon tetrachloride was based upon data obtained from Clifford who determined by the Calcium Chloride method [16] that the solubility of HOH in CCl_4 was 0.013% at 28.5°C. This results in a concentration of 0.0140 M H_2O in CCl_4 when saturated at 28.5°C. Saturated solutions of H_2O in CCl_4 were prepared by adding 30 lambda of HOH to 100 ml of CCl_4 which gives a concentration of 0.01665 M at 25°C.

D. SPECTRA OF HCl-HOH IN CARBON TETRACHLORIDE

Difficulty in keeping HCl-CCl_4 solutions dry led to investigations of the HCl-H_2O-CCl_4 system. Initial study was directed toward the identification of an absorption band due to the hydronium ion based on the assumed reaction:

$$HOH + HCl \rightleftharpoons H_3O^+ + Cl^-$$

A hydrated form of HCl was also suspected:

$$HOH + HCl \rightleftharpoons HCl \cdot H_2O$$

Bubbling HCl gas through dried CCl_4 for about a minute produced an approximately 0.05M solution. Addition of H_2O-saturated CCl_4 solutions in varying proportions to the HCl-CCl_4 solution produced the spectra shown in Fig. 6. The ratio of HCl to H_2O ranged from 4:1 to 1:3 in the given spectra. The concentration of HCl in CCl_4 solution was not kept constant in this series of analyses.

Analysis of the spectra of the 3-component system failed to show a satisfactory absorption band between 2500 cm^{-1} and 4000 cm^{-1} which could be attributed to either H_3O^+ or hydrated HCl. A suspicious band occurring at 3540 cm^{-1} and an accompanying broad band between 3300 cm^{-1} and 3500 cm^{-1} turned out to be absorptions due to white spots being formed on the BaF_2

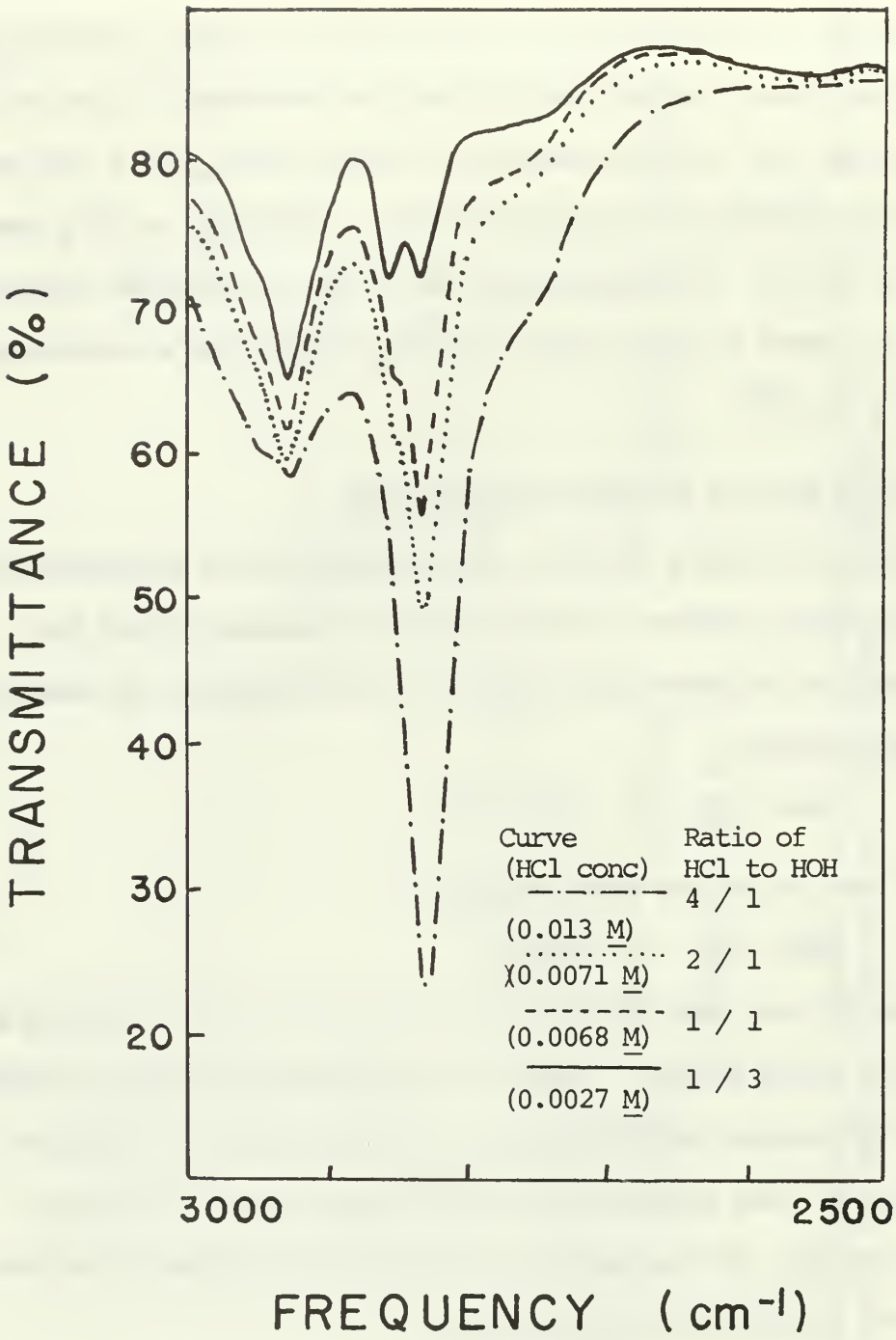

Figure 6. Spectra of HCl-HOH in carbon tetrachloride solutions, with varying proportions of HCl to HOH. Concentration of HCl ranged from 0.013 M to 0.0027 M in the HCl to HOH ratios indicated.

windows. These spots were relatively easy to clean but recurred when HCl and H_2O were in the CCl_4 solvent at the same time. The spots were readily soluble in H_2O and some form of a hydrated compound was suspected.

With the NORELCO X-ray diffractometer, filtered copper K-2 radiation was used to identify the contamination by analysis of the diffraction patterns. The contamination was most probably $BaCl_2 \cdot 2H_2O$.

Another analysis involved keeping the concentration of HCl relatively constant while adding small amounts of H_2O. Varying the relative concentrations of HCl to H_2O in (use of the Lambda pipette) solutions produced some interesting results. It was noted that the intensity of the HCl absorption peak remained relatively constant for a given concentration independent of the HOH concentration. The intensity of the ν_3 fundamental frequency of HOH did not follow the Beer-Lambert Law when HCl was present in solution. This effect indicated that rotational-vibrational freedom of HOH in solution was altered by the presence of HCl although there was no apparent association or interaction between the two. Water was apparently involved in some type of reaction. Keeping the concentration of HCl relatively constant while varying the HOH concentration was quite workable but the reverse was more difficult.

When the HCl concentration became quite dilute (<0.005 M) an absorption peak at 2850 cm^{-1} appeared and the band shoulder corresponding to the R-branch of gaseous HCl assumed greater intensity (Fig. 6). The observed bands are again suspected to be due to the additional interaction of HCl-H_2O although the nature of interaction is not evident.

The analysis of the spectra was extended into the 1500 to 2500 cm^{-1} region quite accidentally when the automatic scanning feature of the spectrometer was not stopped at 2500 cm^{-1}. Two weak but observable peaks

were observed at 1710 cm^{-1} and 1765 cm^{-1} for the three-component system. (Fig. 7) These peaks were suspected to be due to the hydronium ion or a new species and their disappearance in the spectra for DCl solutions supports this hypothesis. Additional investigations need to be conducted to locate the D_3O^+ absorption bands in another solvent because of strong solvent absorption of CCl_4 beyond 1500 cm^{-1}.

Analysis of the absorption spectra of the HCl-HOH in CCl_4 also yielded significant results regarding the extent of interaction between hydrochloric acid and water in the non-polar solvent. Based on the assumption that hydronium ion is formed according to the equation:

$$HCl + HOH \rightleftarrows H_3O^+ + Cl^-$$

the equilibrium constant for the reaction in carbon tetrachloride

$$K_{eq}(CCl_4) = \frac{[H_3O^+]\ [Cl^-]}{[HCl]\ [HOH]}$$

was computed to be approximately equal to 10^{-3} at 25°C. This result confirms the relatively small interaction between the two compounds in carbon tetrachloride as compared to that in aqueous solution.

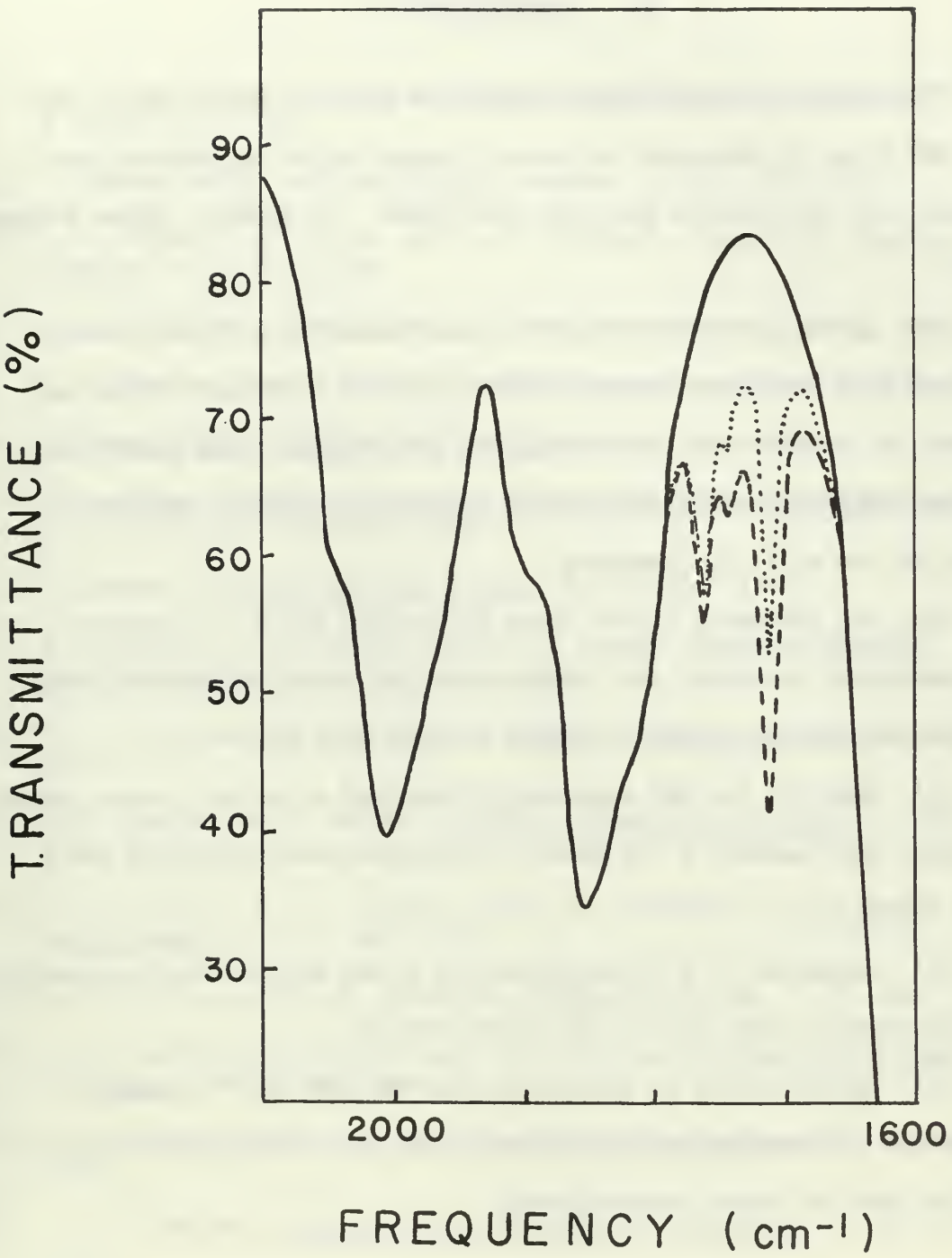

Figure 7. Infrared spectra of HCl-HOH in carbon tetrachloride solution in the 1600-2100 cm^{-1} range. The peaks at 1710 cm^{-1} and at 1765cm^{-1} are probably due to the hydronium ion absorption. The solid line curve is the pure solvent spectrum; the dotted line is the 4:1 ratio of HCl to HOH in solution; the dashed line is that of the 1:3 ratio of HCl to HOH in solution.

IV. CONCLUSIONS

The factors discussed above indicated that the HCl molecule and the HOH molecule dissolved in carbon tetrachloride interacted very weakly with the solvent and with each other. In summary, these factors were:

(a) It has been shown that HCl when dissolved in a non-polar solvent such as carbon tetrachloride displayed a Q-branch which was absent in its infrared spectrum in its pure state. This absorption further indicated an interaction of the solute with the solvent as shown by the shift in frequency.

(b) The frequency shifts shown by the HCl and DCl in carbon tetrachloride indicated the hydrogen bonding which occurred with HCl in the solvent is similar in nature as that with DCl.

(c) Both HCl and HOH separately dissolved in carbon tetrachloride followed Beer-Lambert's Law when the solutions were dilute (0.001 M to 0.020 M).

(d) Formation of a hydronium ion or a new species was suggested by the absorption peaks at 1710 cm^{-1} and 1765 cm^{-1}.

(e) Appearance of an absorption peak at 2850 cm^{-1} strongly suggested interaction in very dilute (less than 0.001 M) solutions of HCl and HOH in carbon tetrachloride.

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13. ABSTRACT

The infrared vibrational-rotational spectra of hydrochloric acid and water dissolved together in carbon tetrachloride were obtained and compared with the spectra of hydrochloric acid in carbon tetrachloride and of water in carbon tetrachloride separately. Spectra of extremely dilute solutions of the two solutes in the non-polar solvent were analyzed for nature and extent of solvent-solute interaction. Dilution behavior was investigated by the use of the Beer-Lambert Law. Possible resulting species were also investigated.

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hydronium ion						